

Stereolithography Apparatus for Free Form Fabrication of Ceramics

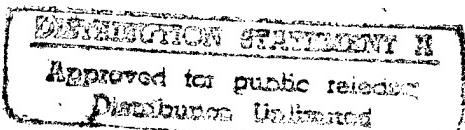
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Office of Naval Research

**ONR Scientific Officer: Steven G. Fishman
01 January 1995- December 31, 1996**

**Final Technical Report
August 25, 1997**

**Principal Investigator: John W. Halloran
Department of Materials Science and Engineering
University of Michigan
Ann Arbor, MI 48109-2136**



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Stereolithography Apparatus for Free Form Fabrication of Ceramics

Final Technical Report

Abstract

During this grant we installed a 3D Systems SLA 250/40 stereolithography apparatus, and used it to directly fabricate ceramic green bodies from ultraviolet (UV) curable solutions which contain dispersed ceramic powders. We emphasized SLA of alumina as a structural ceramic and for investment casting cores. Viscosity was reduced with improved dispersion. The quality of the prototypes was improved by determination of appropriate build styles, and by optimization of photoinitiators and photoresins. The latter were studied by photo-differential scanning calorimetry, and photo-rheology.

ONR Contract Information is summarized below

Contract Title: StereoLithography Apparatus for Free Form Fabrication of Ceramics

Performing Organization: University of Michigan

Principal Investigator: John W. Halloran

Contract Number: N00014-95-1-0527

R & T Project Number: cer9501---01

ONR Scientific Officer: Steven G. Fishman

Total Contract Value: \$ 156, 121 from ONR

and \$235,266 for equipment from DARPA

Contract Start Date: 1 January, 1995

Contract End Date: December 31, 1996

Scientific Research Goals

We were to develop a method for free form fabrication of ceramic green bodies using stereolithography of fluid suspensions of ceramic powders in photopolymerizable monomer solutions. With this method, highly complex ceramic objects are built from aluminum oxide with existing commercial stereolithography hardware and software, to allow ceramic prototypes to be made on these machines without modifications. This was successful, and the SLA apparatus is routinely used to build dense ceramics with excellent shape fidelity.

Significant results of the program

This project follows a previous ONR grant (N00012-93-1-0302 which supported the PhD thesis research of Michelle Griffith. Griffith developed a method to conduct free form fabrication of

ceramics by building ceramic green bodies in a stereolithography apparatus (SLA), using a photo-curable ceramic suspension in place of the resin. Griffith demonstrated the concept in an SLA machine in the development lab of 3D Systems, Inc.

The present grant provided funds for Michigan to acquire an SLA 250/40 stereolithography apparatus, and funded the first two years of the PhD thesis of G. Allan Brady. Brady has emphasized aluminum oxide, and has focused on building complex objects by SLA. This has involved significant improvements of the ceramic resin and SLA technique. At the end of the grant (December 1996), Brady was unable to build complex multilayer objects, due to several problems. Many of these problems were overcome shortly after the grant, during Spring and Summer 1997. The progress can be illustrated by example parts.

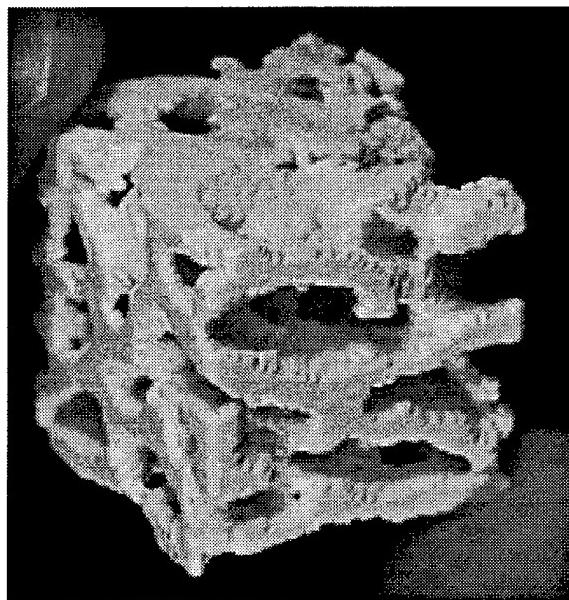


Figure 1 An aluminum oxide model of human trabecular bone tissue, built 8.5 times life size. The ceramic model is about 20 mm square.

Figure 1 is an aluminum oxide model of human trabecular bone tissue. We present it as an example of a very complex object with very detailed interconnecting solid and void phase. It was built from a micro-Computed Tomography (CT) file obtained by S.J. Hollister and converted to a STL file with 626,000 facets. We built this model out of SLA epoxy for biomechanical experiments¹, but also find it a useful model for ceramic stereolithography. This model weighed 18.4 gm and had a solid volume of 7.1 ml. It

¹ P.K. Zysset, A.L. Marsan, T-M Chu, R.E. Guldberg, J. W. Halloran and S.J. Hollister, "Rapid Prototyping of Trabecular Bone for Mechanical Testing", in press, 1997

required a 40 hour build time. Post treatment involved rinsing the object to remove un-cured resin, slow heating to 600°C to burn away the acrylate binder, and sintering to 1600°C. The ceramic is fully dense.

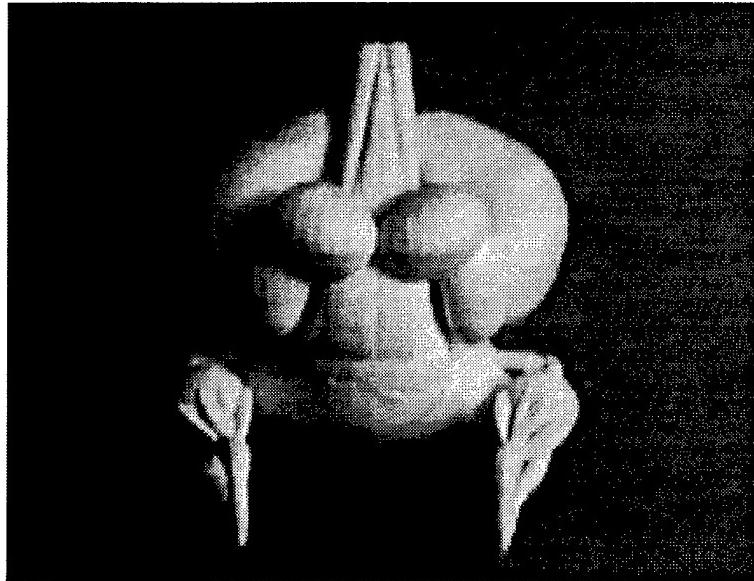


Figure 2 An aluminum oxide sculpture built from a file by sculptor Michael Rees.

Figure 2 is an untitled sculpture by Michael Rees, a sculptor who is pioneering SLA art. We received an STL file from Rees, and initiated the build in early June. This is the largest object we have yet built. Its volume is 58 ml, and it weights 118 gm in the sintered condition. The SLA built required 248 hours, during which the SLA machine ran unattended and continuously.

These and other SLA ceramic objects can be seen at the web site: msewww.engin.umich.edu/People/Halloran/SL/ceramicsla.html, which also includes copies of all recent papers and presentations.

Much of the progress resulted from a detailed study of the curing of the ceramic photoresin. To do this, Brady built a photo-rheology apparatus by adapting a Bohlin CS-50 Rheometer so it could monitor the properties of the ceramic resin as a function of UV dose. He also converted a Perkin-Elmer DSC-7 apparatus for photo-differential scanning calorimetry. This information allowed him to optimize the acrylate system and the photoinitiator system. This work is described in more detail in the paper 1Stereolithography of Ceramic Suspensionsx, in the Appendix.

Brady is continuing his research, and writing his PhD thesis. This should be completed by November 1997, and will result in several additional publications. Below is a list of papers already finished.

List of Publications/Reports/Presentations

1. Papers Published in Refereed Journals

G. Allen Brady and John W. Halloran, 1Stereolithography of Ceramic Suspensionsx, to appear in the Proceedings of the 6th International Conference on Rapid Prototyping and Mfg in Nottingham , July 1-3, 1997 in the Journal of Rapid Prototyping

2. Non-Refereed Publications and Published Technical Reports

G. Allen Brady, T-M. Chu, and J.W. Halloran, xCuring Behavior of Ceramic Resin for Stereolithographyx, in*Solid Free Form Fabrication Proceedings* , J.J. Beaman, J.W. Barlow, D.L. Bourell, and R.H. Crawford, editors, 1996 SFF Symposium, Austin Texas, pp 403-410 [1996]

Appendix

This Appendix has a preprints of:

G. Allen Brady and John W. Halloran, 1Stereolithography of Ceramic Suspensions, to appear in the Proceedings of the 6th International Conference on Rapid Prototyping and Mfg in Nottingham , July 1-3, 1997 in the Journal of Rapid Prototyping

Stereolithography of Ceramic Suspensions

G. Allen Brady and John W. Halloran
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The University of Michigan, Ann Arbor, MI. 48109

Abstract

Rapid prototyping of ceramics are accomplished with stereolithography by using an SLA machine to build the ceramic green from a UV- curable suspension of ceramic powders - a "ceramic resin". Objects are later sintered in a separate furnace to complete the process. Aluminum oxide resins based upon hexane diol diacrylate are characterized for curing behavior by photo-rheology and differential photo calorimetry with a UV lamp, and with a HeCd laser using "windowpanes", single strings, and walls

This research was supported by the US Office of Naval Research under grant N00014-95-1-0527, through Dr. S. Fishman and R. Wachter

Introduction

Free form fabrication of ceramics can be accomplished by stereolithography (SLA) by using as the "resin" a suspension of ceramic powder in a UV-curable medium¹. We have explored several "ceramic resins" for aluminum oxide, silicon dioxide, and hydroxyapatite in acrylate and aqueous media². These very concentrated suspensions of ceramic powder are highly turbid due to scattering³ of the UV by the ceramic particles, but can have a useful depth of cure.

This paper concerns the behavior of a simple resin consisting of alumina powder suspended in hexane diol diacrylate (HDDA). We examine the cure depth as a function of dose from the HeCd laser in the SLA 250. The kinetics of the polymerization reaction are examined by thermal analysis, using a UV radiation from a Hg-vapor lamp in a differential photo calorimeter (DPC). Gelation is characterized by monitoring the storage modulus in a rheometer during irradiation with a UV lamp (photo-rheometry).

Experimental Procedures

Ceramic Resin

The ceramic resin consists of 50 volume% Al_2O_3 powder^a in 1,6 hexanediol diacrylate^b (HDDA), stabilized to create a fluid suspension using a quaternary ammonium acetate^a dispersant. The photoinitiator was 1-hydroxy-cyclohexyl phenyl ketone^d at a concentration of 0.5 wt%. The ceramic resin is stable against

^a RCHP DBM, Malokoff Industries, Malokoff, TX, USA, median particle size 0.3 microns

^b Photomer 4017, Henkel Corp., Kankakee, IN, USA

^c Emcol CC-55, Organics Div., Witco Corp., NY, NY USA

^d Irgacure 184, Ciba-Geigy, Ciba Additives, Tarrytown, NY USA

sedimentation with a nearly Newtonian viscosity of 200 mPa-s, which is similar to a standard commercial epoxy resin^e. Stereolithography experiments were conducted in a 3D Systems, Inc. SLA 240/40 apparatus, modified with a mini-vat² which allows the apparatus to function with only 2 liters of experimental resin. The behavior of the diacrylate-based ceramic resin is compared with the commercial Cibatool SL5170 epoxy resin, typically used with the SLA 250.

Photo-Rheology and Differential Photo Calorimetry

Photo-rheological measurements were taken with a Bohlin CS-50 rheometer^b in a plate-plate configuration. The bottom plate of the rheometer was made from UV - transparent silica glass. Ultraviolet radiation at a dose rate of 2.5 mJ/cm²sec was introduced through the silica glass plate from a Pen-Ray lamp^c at a rate of 2.5 mJ/cm²sec. This lamp emits the spectrum characteristic of a medium pressure Hg lamp, with the largest emission at 365 nm.. The apparatus is illustrated in Figure 1. The excitation conditions were oscillation at 10 Hz at an applied shear stress of 500 Pa and an equilibrium time of 30 seconds. For each measurement, the UV lamp was turned on at about 15.7 seconds and remained on throughout the test. All the data presented here were collected with a 0.2 mm gap thickness.

This technique monitors the transition between viscous liquid ($\eta \sim 200$ mPa-s) to fully cured solid ($G' \sim 1$ MPa) during oscillation in a plate-plate geometry. Otsubo et al^d describes a similar plate-plate oscillatory viscometer for measuring photosensitive polymers. The purpose of this measurement technique is to probe the change of liquid-solid transition during uv-curing of these ceramic suspensions.

The Differential Photo Calorimetry experiments were conducted with a Perkin-Elmer DSC-7 apparatus^d modified to introduce UV radiation from the Pen-Ray lamp at 7.3 mJ/cm²sec. The experiments were run at 40°C with a sample size of 5-10 mg, for which isothermal conditions could be satisfactorily maintained by the DPC. The data reported in this paper are the average of five runs. The sample chamber is contained in a flowing N₂ atmosphere to eliminate the effect of oxygen inhibition.

Results and Discussion

The results from DPC appear in Figure 3 as plots of heat flow vs. dose. The pure HDDA (0% aluminum oxide) reacts very rapidly at a low dose, reaching a peak heat evolution rate of 120 J/gm-sec. Addition of 50 vol% aluminum oxide (or 83 wt%) greatly moderates the rate of the reaction, so that the peak heat evolution rate drops to 3.6 J/gm-sec. This effect appears to be greater than a simple dilution with inert filler, so probably represents the effect of the highly scattering aluminum oxide

^eCibatool SL 5170, Ciba-Geigy Corporation, Ciba Specialty Chemicals, Performance Plastics Div., Lansing, MI, USA

^b Bohlin-Reologi, Lund, Sweden

^c Pen-Ray Lamp 11SC-1L, UVP, Inc., Upland, CA, USA

^d Perkin-Elmer Corp., Norwalk, CT USA

powder. The reactivity of the aluminum oxide-HDDA resin is slower than the SL5170 epoxy system.

The DPC data can be expressed as conversion of monomer vs. dose by assuming that the unfilled resins are fully reacted. This data is presented in Figure 4. Note that unfilled HDDA (0% aluminum oxide) is 75% reacted at a dose at which the 50% aluminum oxide-HDDA resin and the epoxy are only 3-5% reacted. The aluminum oxide-filled resin is comparable in behavior up to about 70% conversion. Beyond that point the curing of the ceramic resin is greatly retarded. About 15% of the HDDA monomer in the aluminum-oxide system remains unreacted at the end of the experiment.

Photo rheological measurements provide in-situ monitoring of UV-curing films. As the resin cures to a gel point, the storage modulus increases over many orders of magnitude. This change is easily detected under oscillating stress conditions applied by the rheometer. The dose required to begin curing, or cure dose, is measured sudden change in the storage modulus. The cure dose varies predictably with the gap thickness, following a Beer-Lambert type relation where Log(cure dose) varies linearly with gap thickness. Other investigators⁵ have observed such a relation between cure dose and the depth of cured films. From this we infer a D_p of 156 microns and an E_c of 21 mJ/cm² for the 50% aluminum oxide resin.

The curing of the 0.2 mm thick layer in the photo rheometer is shown in Figure 5, which shows the storage modulus as a function of dose. The two HDDA-based resins display a sudden increase in storage modulus at a dose of about 60-80 mJ/cm². The storage modulus reaches a limiting value around 90 MPa after a dose of about 100 mJ/cm². The onset of curing for the epoxy SL 5170 is also about 80 mJ/cm², but the storage modulus builds quite gradually with dose, in what appears to be a two stage process. A dose of 800 mJ/cm² is required to achieve a limiting value of modulus of about 8.8 MPa. Measurements of the loss modulus and shear viscosity vs. dose also show a sudden onset occurring at the same cure dose observed with the storage modulus. There is essentially no change in viscosity of the resins at exposures smaller than the cure dose.

While the storage modulus vs. dose is similar for the HDDA resin and the 50% aluminum oxide-HDDA resin, there is a large difference in the modulus vs. monomer conversion. Nearly 70% of the monomer in the unfilled HDDA is reacted at the cure dose, so a large fraction of polymer forms before gelation. The aluminum oxide-filled HDDA has about the same cure dose, but only about 2% of the monomer has reacted at the cure point. Very little polymer has formed at the point of gelation, certainly not enough for polymeric gelation.. Recall that the 50% aluminum oxide-HDDA resin is quite concentrated colloidal suspension of submicron powders, kept in dispersion by the action of the dispersant. Without the dispersant, HDDA containing 50 volume percent aluminum oxide powder is a stiff paste-- a colloidal gel. Thus the initial curing of the ceramic resin might be by a photochemically-induced colloidal gelation.

The penetration depth and the critical exposure energy of the 50% aluminum oxide-HDDA resin was characterized on the SLA 250 using the “windowpanes” technique. The thickness of windowpanes vs. dose appears in Figure 7, from which the build parameters $D_p = 62$ microns and $E_c = 33 \text{ mJ/cm}^2$ can be derived. These values are quite different from the typical SL 5170 epoxy resin, so building with this particular ceramic resin requires much longer exposures.

Stereolithography builds objects from ‘strings’ (cured lines) defined as the scanning laser beam traverses the surface of the resin. The cross section of a string in a non-scattering resin is a bullet-shaped⁶ parabolic curve, typically much deeper than it is wide. Cured line profiles in the highly-scattering ceramic resin are much flatter. Figure 7 is an optical micrograph of a single string in this aluminum oxide resin. It is about 230 microns deep and 520 microns wide, flattened by attenuation of the forward beam by ceramic filler, and widened by side scatter. The edges are slightly scalloped, a consequence of side scatter of the UV. These effects are sensitive to the difference in refractive index of the filler and the medium, so are different for other ceramic powders and monomers.² A vertical section with these flattened bullets has scalloped sides, as shown in Figure 8. This is an optical micrograph of a vertical wall created from a sequence of stacked borders. Note that the side scattering creates scalloped side projections about 50 microns wide.

After the SLA build is completed, the object is a ceramic “green body” with the photocured resin as its binder. The polymeric binder is removed by heating in air at $1^\circ\text{C}/\text{min}$, a typical rate for binder removal. By about $600\text{-}800^\circ\text{C}$ in air, all of the polymer residue and char is removed. This grade of aluminum oxide powder begins to sinter above 1200°C , and sinters to a pore-free state at 1600°C . Figure 9 is a scanning electron micrograph of the surface of the sintered alumina, showing excellent sintering of the ceramic.

Conclusions

Pore free aluminum oxide ceramics can be built by stereolithography using a concentrated suspension of ceramic powder in a UV-curable medium.

The photocuring of the ceramic resins has been characterized with photo-differential scanning calorimetry and photo-rheometry. Photo-DSC reveals that the aluminum oxide filled HDDA system polymerizes much more slowly than simple HDDA. Photo-rheometry shows that the aluminum oxide filled HDDA system cures at nearly the same dose as the pure HDDA. Gelation occurs at only 2% reaction of the monomer, so onset of rigidity must be due to colloidal gelation, rather than polymeric gelation.

For the aluminum oxide-HDDA resin at this photoinitiator content, penetration depth $D_p = 62$ microns and the critical exposure energy $E_c = 33 \text{ mJ/cm}^2$. Due to scattering of the UV radiation by the ceramic particles, the laser drawn strings for this ceramic resin are flattened, more than twice as wide as they are deep. Vertical walls are therefore scalloped.

References

- 1) M.L. Griffith and J. W. Halloran (1996) Free Form Fabrication of Ceramics via Stereolithography J. American Ceramic Soc. 79 [10], 2601-08
- 2) G. Allen Brady, T-M. Chu, and J.W. Halloran (1996) Curing Behavior of Ceramic Resin for Stereolithography in **Solid Free Form Fabrication Proceedings** J.J. Beaman, J.W. Barlow, D.L. Bourell, and R.H. Crawford (eds) 1996 SFF Symposium, Austin Texas, pp 403-10
- 3) M.L. Griffith and J. W. Halloran (1997) Scattering of Ultraviolet Radiation in Turbid Ceramic Suspensions **J. Applied Physics** (in press)
- 4) K. Watanabe, T. Amari and Y. Otsubo (1984) Dynamic Viscoelastic Measurements of Photosensitive Polymers **J. Applied Poly. Sci.** 29, 57-66
- 5) Y. Otsubo, T. Amari and K. Watanabe (1984) Rheological Behavior of Epoxy Acrylate Prepolymer during UV-Curing **J. Applied Poly. Sci.** 29, 4071-80
- 6) P.F. (1992) **Rapid Prototyping and Manufacturing: Fundamentals of Stereolithography**, Soc. of Manufacturing Engineers, Dearborn, p. 87

Captions for Illustrations

Figure 1: Schematic of Photo Rheology measurement

Figure 2: Schematic of Differential Photo Calorimetry measurement

Figure 3: Heat flow vs. dose from DPC measurements of: 1) HDDA (0% aluminum oxide); 2) 50 volume percent aluminum oxide in HDDA; and 3) Cibatool SL5170

Figure 4: Percent conversion of monomers vs. dose for: 1) HDDA (0% aluminum oxide); 2) 50 volume percent aluminum oxide in HDDA; and 3) Cibatool SL5170

Figure 5: Photo-reological measurements comparing storage modulus vs. dose for: 1) HDDA (0% aluminum oxide); 2) 50 volume percent aluminum oxide in HDDA; and 3) Cibatool SL5170

Figure 6: Depth of cure vs. UV laser dose from SLA windowpanes experiment

Figure 7: Cured line profile for SLA laser-cured line for 50 volume percent aluminum oxide in HDDA

Figure 8: Vertical wall built from stacked borders for SLA laser-cured 50 volume percent aluminum oxide in HDDA

Figure 9: High magnification SEM micrograph of sintered aluminum oxide

¹ M.L. Griffith and J. W. Halloran (1996) Free Form Fabrication of Ceramics via Stereolithography **J. American Ceramic Soc.** 79 [10], 2601-08

² G. Allen Brady, T-M. Chu, and J.W. Halloran (1996) Curing Behavior of Ceramic Resin for Stereolithography in **Solid Free Form Fabrication Proceedings** J.J. Beaman, J.W. Barlow, D.L. Bourell, and R.H. Crawford (eds) 1996 SFF Symposium, Austin Texas, pp 403-10

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⁵ Y. Otsubo, T. Amari and K. Watanabe (1984) Rheological Behavior of Epoxy Acrylate Prepolymer during UV-Curing **J. Appl. Pol. Sci.** 29, 4071-80

⁶ P.F. Jacobs (1992) **Rapid Prototyping and Manufacturing: Fundamentals of Stereolithography**, Soc. of Manufacturing Engineers, Dearborn, p. 87

Bohlin CS-50 Rheometer
Oscillating Plate-Plate Geometry

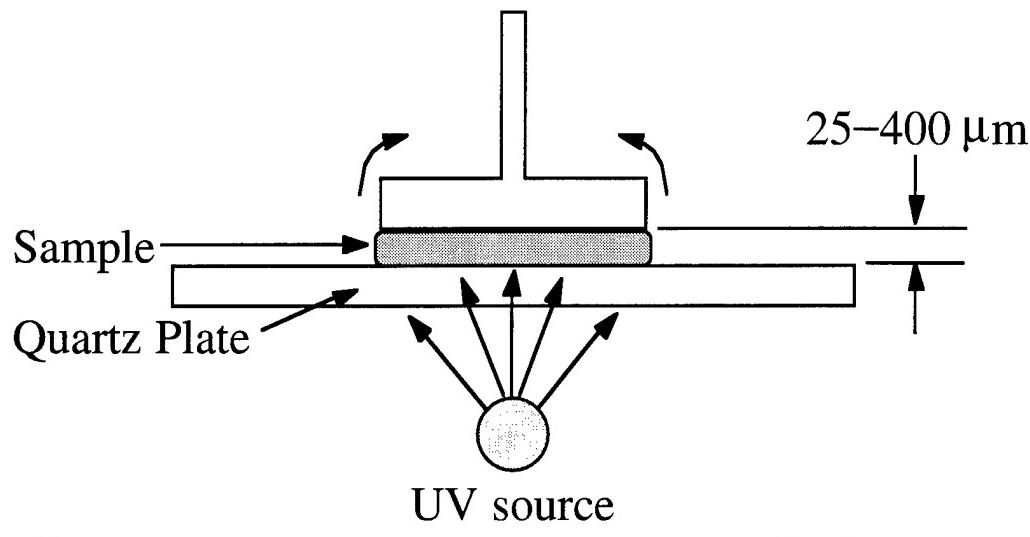


Figure 1 Schematic of Photo Rheology measurement.

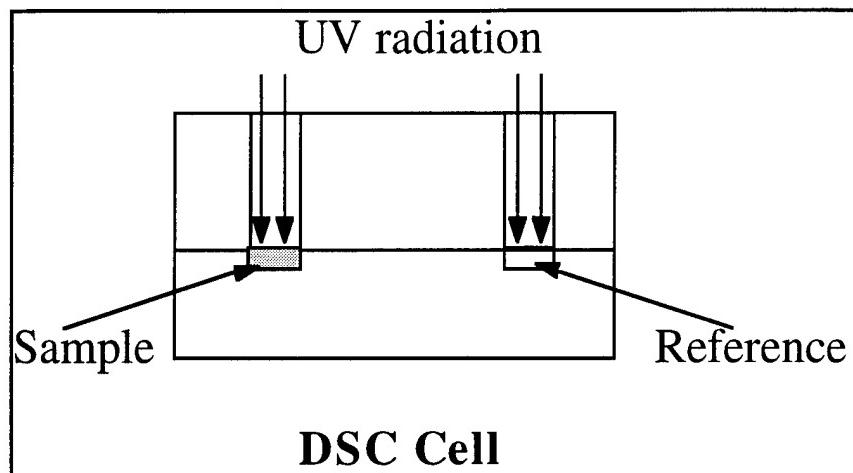


Figure 2 Schematic of Differential Photo Calorimetry measurement.

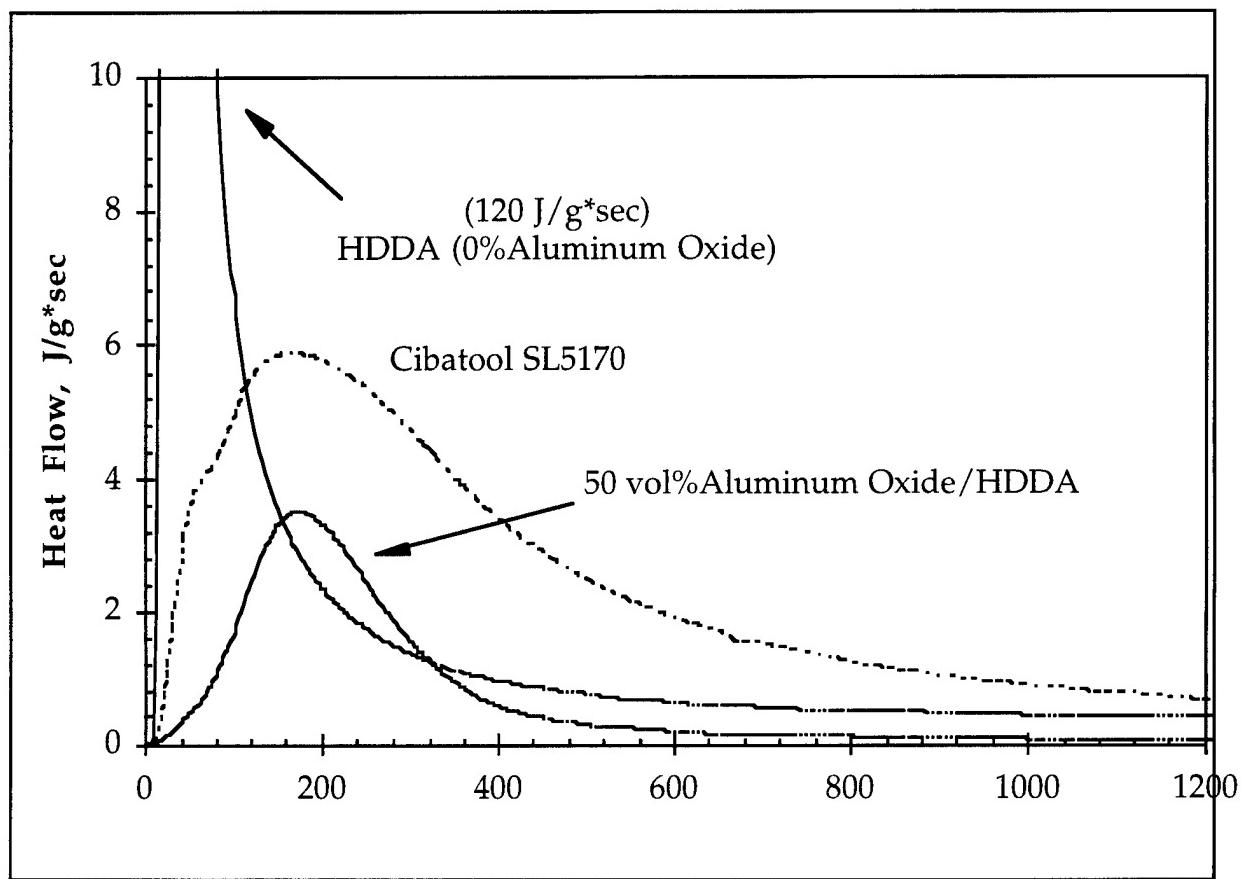


Figure 3 Heat Flow measurement from DPC of 1) HDDA (0% Aluminum Oxide), 2) 50 vol% Aluminum Oxide/HDDA and 3) Cibatool SL5170.

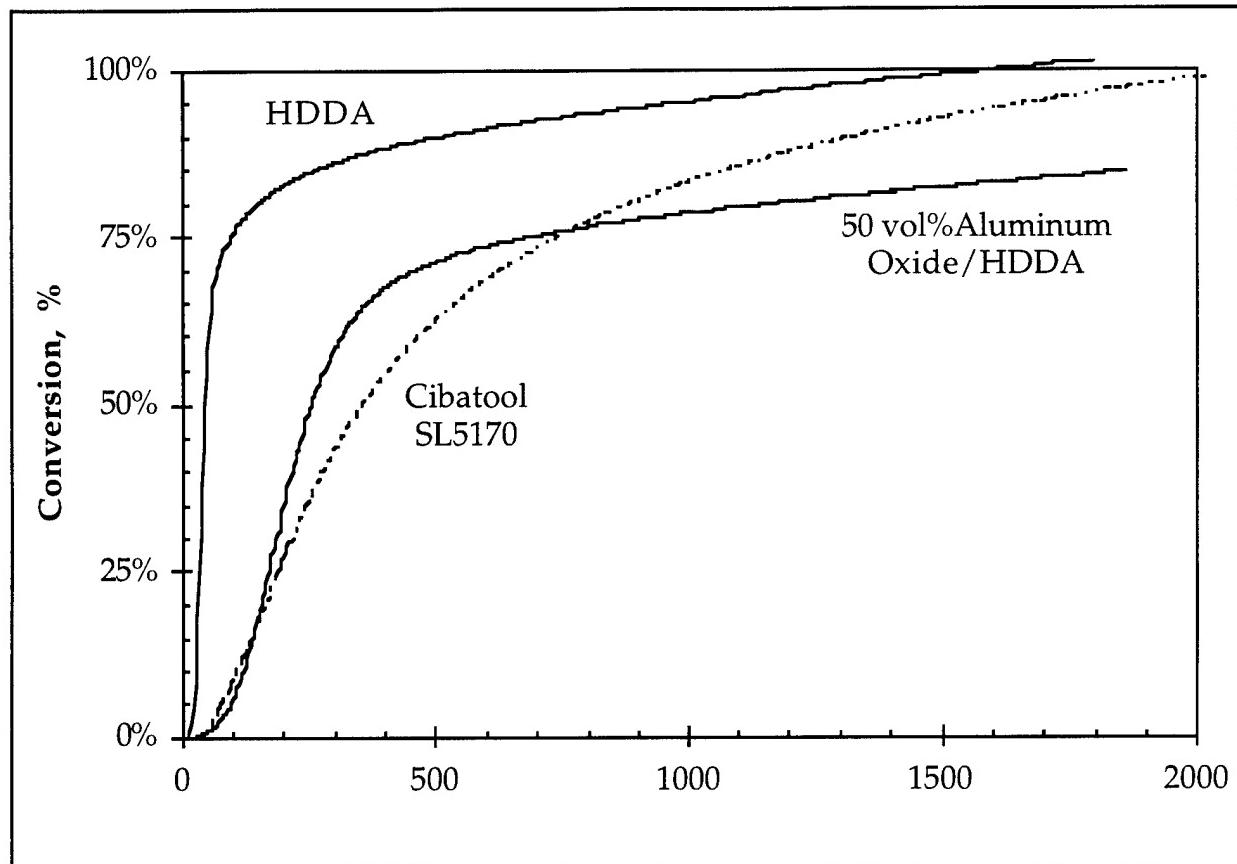


Figure 4 Percent conversion vs. dose from DPC comparing 1) HDDA (0% Aluminum Oxide), 2) 50 vol% Aluminum Oxide/HDDA and 3) Cibatool SL5170.

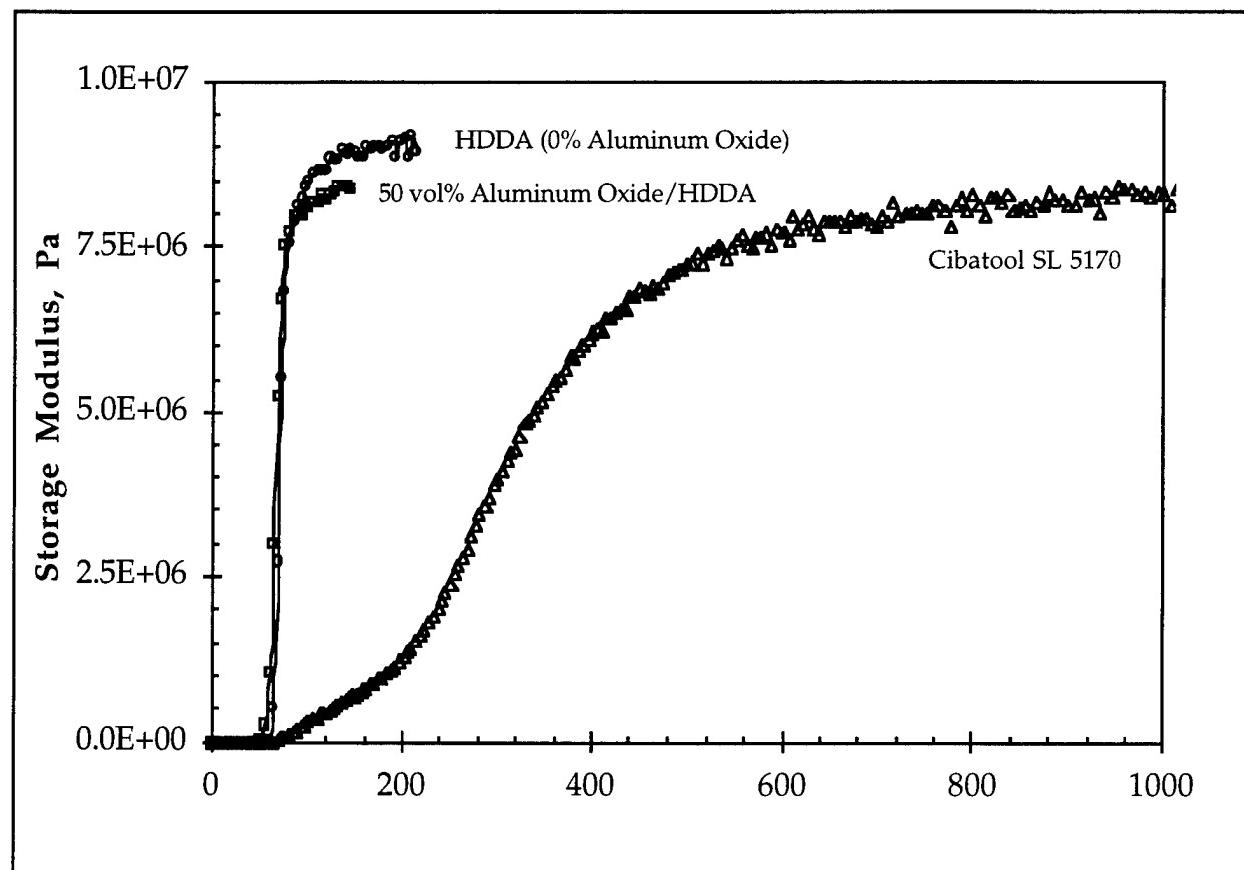


Figure 5 Photorheology measurement of Storage Modulus vs. Dose for 1) HDDA (0% Aluminum Oxide), 2) 50 vol% Aluminum Oxide/HDDA and 3) Cibatool SL5170.

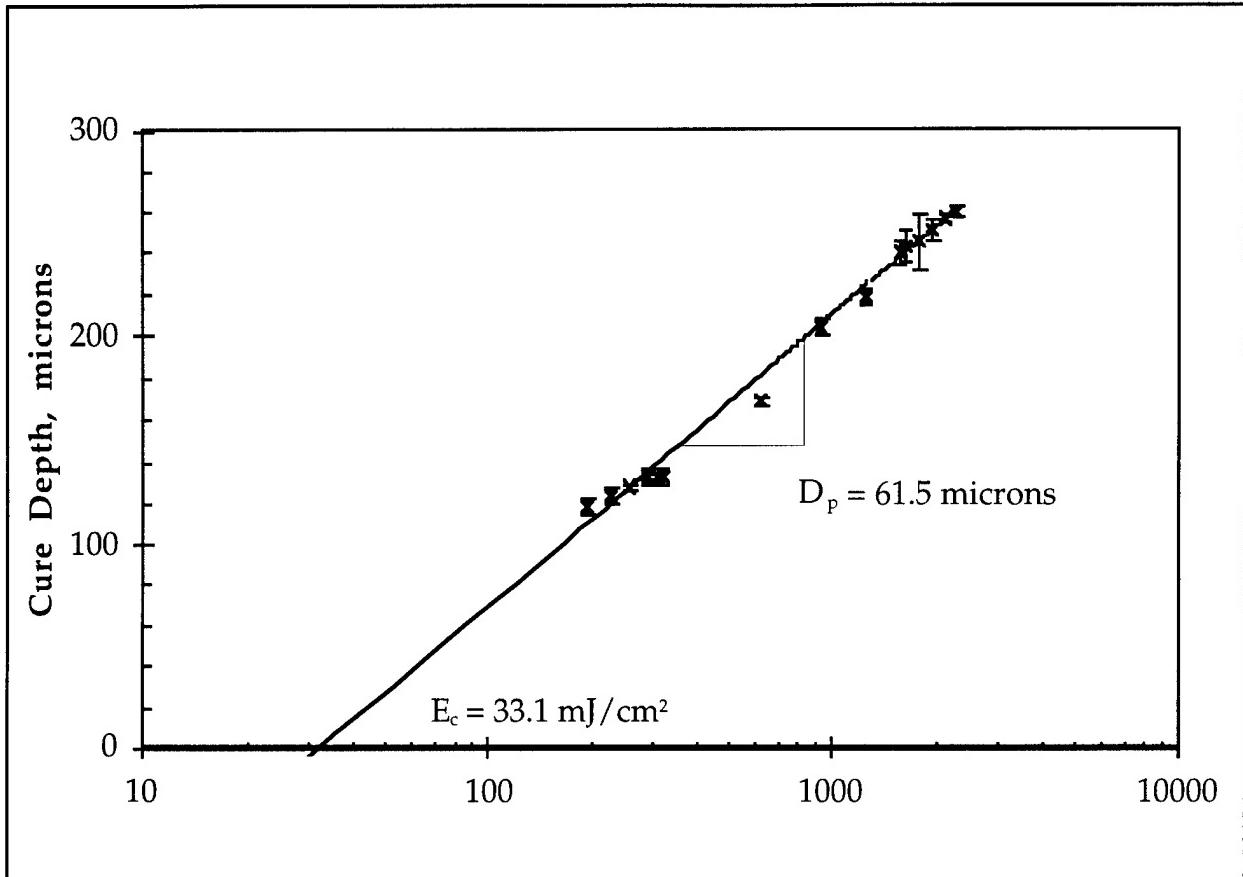


Figure 6 Cure depth vs. dose from Windowpane diagnostic parts fabricated from 50 vol% Aluminum Oxide/HDDA.

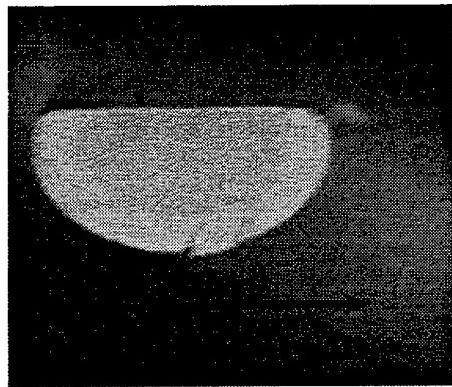


Figure 7 Cured line profile of 50 vol% Aluminum Oxide/HDDA.

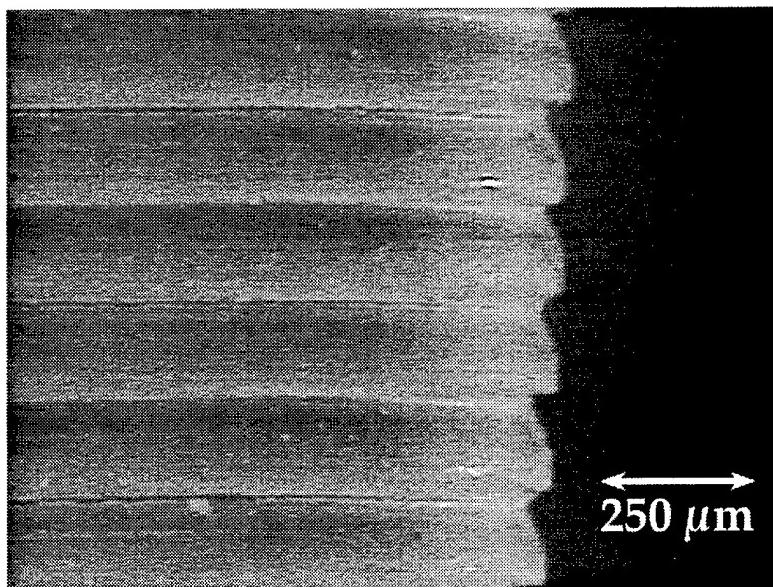


Figure 8 Vertical wall of stacked borders built on an SLA 250 from 50 vol% Aluminum Oxide/HDDA.

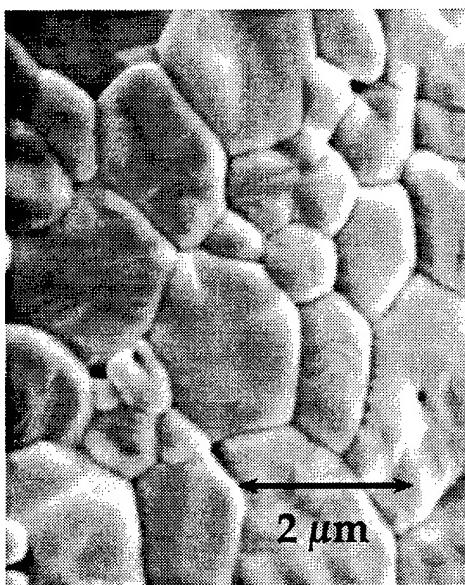


Figure 9 High magnification SEM of sintered Aluminum Oxide.

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